DETECTION OF ELEMENTS IN SLIMMING TEA PRODUCT BY USING LASER INDUCED BREAKDOWN SPECTROSCOPY

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To the people who loved me

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ABSTRACT

Laser induced breakdown spectroscopy (LIBS) has been applied to determine the element content in slimming tea product. A Q-switched Nd:YAG laser operating at 1064 nm and generating 93.7 mJ per pulses was employed to excite the pellet sample and the fluorescence emission was analyzed via spectrum analyzer. The induced spectral lines were compared with National Institute of Standards and Technology (NIST) database to identify the element. A commercial slimming tea was used as sample to be investigated whereas the natural tea leaves were used as control. The LIBS results showed that both slimming tea and natural tea leaves contain several elements including iron (Fe), sodium (Na), chromium (Cr), cobalt (Co), calcium (Ca), manganese (Mn), magnesium (Mg), silicon (Si), sulfur (S), titanium (Ti), nickel (Ni) and cesium (Cs). However, higher intensity of elements was found in slimming tea spectrum compared to natural tea leaves. The plasma temperatures calculated using Boltzmann plot were found to be 13588.95 K and 12319.50 K for slimming tea and tea leaves samples respectively whereas the electron density for slimming tea and tea leaves samples were 7.46×10^{16} cm^{-3} and 15.91×10^{16} cm^{-3}, respectively. The plasma produced by this LIBS system was found to be in local thermodynamic equilibrium (LTE) condition after evaluating these plasma parameters. Quantitative analysis performed by using inductively coupled plasma mass spectroscopy (ICPMS) proved that the concentration of the elements is higher in slimming tea than in natural tea leaves.

ABSTRAK

Teknik spektroskopi leraian aruhan laser (LIBS) telah digunakan untuk menganalisis kandungan unsur di dalam produk teh pelangsingan. Sebuah laser suis-Q Nd:YAG beroperasi pada 1064 nm dan menjana 93.7 mJ per denyut telah digunakan untuk mengujakan sampel gentel dan pencucuran pendarfluor yang terhasil dianalisis melalui penganalisis spektrum. Garisan spektrum yang terhasil dibandingkan dengan data dari Institut Piawaian dan Teknologi Kebangsaan (NIST) untuk mengenal pasti unsur yang diwakilinya. Teh pelangsingan komersial digunakan sebagai sampel untuk dianalisis manakala daun teh semula jadi telah digunakan sebagai piawai. Hasil LIBS menunjukkan bahawa kedua-dua teh melangsingkan badan dan daun teh semula jadi mengandungi beberapa unsur logam berat termasuk ferum (Fe), natrium (Na), kromium (Cr), kobalt (Co), kalsium (Ca), silikon (Si), mangan (Mn), magnesium (Mg), sulfur (S), titanium (Ti), nikel (Ni) dan cesium (Cs). Walau bagaimanapun, kadar keamatan spektrum bagi teh pelangsingan badan adalah lebih tinggi berbanding daun teh semula jadi. Suhu plasma yang telah dihitung dengan menggunakan kaedah plot Boltzmann ialah 13588.95 K untuk teh pelangsingan badan dan 12319.50 K untuk daun teh semula jadi manakala ketumpatan elektron untuk teh pelangsingan badan dan daun teh semula jadi masing-masing ialah 7.46×10^{16} cm^{-3} dan 15.91×10^{16} cm^{-3}. Plasma yang dihasilkan oleh sistem LIBS ini didapati berada dalam keadaan keseimbangan termodinamik setempat (LTE) selepas menilai parameter plasma. Analisis kuantitatif yang dilakukan menggunakan teknik plasma berganding aruhan spektrometri jisim (ICPMS) telah membuktikan bahawa kadar kepekatan unsur yang terkandung dalam teh pelangsingan badan adalah lebih tinggi berbanding daun teh semula jadi.

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LIST OF SYMBOLS

A_{ki}	-	transition probability
c	-	speed of light
C	-	carbon
Cd	-	cadmium
cm	-	centimeter
Co	-	cobalt
Cr	-	chromium
Cu	-	copper
Cs	-	cesium
E_k	-	upper level energy
eV	-	electron volt
Fe	-	iron
g	-	gram
g_k	-	upper level statistical weight
h	-	Plank constant
I	-	intensity
J	-	joule
k	-	Boltzmann's constant(8.617 × 10-5 eV K-1)
kN	-	kilo Newton
m	-	meter
mA	-	milliampere
Mg	-	Manganese
mJ	-	millijoule
µm	-	micrometer
mm	-	millimeter
Mn	-	manganese

ms	-	millisecond
Na	-	sodium
Ni	-	nickel
n	-	number density of emitting species
nm	-	nanometer
N_e	-	electron density
N(T)	-	total density
ppm	-	part per millions
S	-	slope of the calibration curve
s	-	second
σ	-	standard deviation of the background
S	-	Sulfur
Si	-	Silicon
T	-	temperature
Ti	-	Titanium
U	-	partition function
V	-	volt
λ	-	wavelength

LIST OF ABBREVIATIONS

AAS	-	Atomic Absorption Spectroscopy
AES	-	Atomic Emission Spectroscopy
FDA	-	Food and Drug Administration
FOC	-	Fiber Optic Cable
HPLC	-	High Performance Liquid Chromatography
ICP	-	Inductively Coupled Plasma
ICPMS	-	Inductively Coupled Plasma Mass Spectroscopy
LIBS	-	Laser Induced Breakdown Spectroscopy
LOD	-	Limits of Detection
LTE	-	Local Thermodynamic Equilibrium
MPC	-	Maximum Permissible Concentration
Nd:YAG	-	Neodymium-Doped Yttrium Aluminum Garnet
NIST	-	National Institute of Standards and Technology

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

In the past few years, several research attempts have been made in materials and fabrication nano devices, especially in electronic and optical application. The enhancements are rapidly growing as to fulfil current industry demand in various sectors. As its nano scale size promises advantage including costing, higher efficiency in performance and ability to allow automation tasks in limited physical accessible, the demands have been spread to various fields. Since future industrial growth may started to look into low scale dimension as highly consideration for alternatives technology, an advance exploration has to be conducted in order to meet future needs. Thus, there are theory predictions on replacement of micro device with nano device within 15 years time from year 2000 due to fundamental and economic reasons [1]. Micro-electro-mechanical System (MEMS) and Nano-electro-mechanical System (NEMS) is one of the innovation attempted in order to meet current demand.

The unique and fascinating properties of nanostructured materials have triggered tremendous motivation among scientists to explore the possibilities of using them in technological applications. In particular, the electronic and optical properties of nanostructured materials have been of interest because of their potential applications in the fabrication of nano electronic and optoelectronic devices [2–7]. Nanomaterial research is a field that takes a materials science-based approach on nanotechnology. It studies materials with morphological features on the nanoscale, and especially those that have special properties stemming from their nanoscale dimensions. The nanoscale usually refers to structures with a length scale applicable to nanotechnology, usually cited as 1–100 nanometers [8]. The nanoscale is the size at which fluctuations in the averaged properties begin to have a significant effect on the behavior of a system. Thus, sometimes nanoscale is marked as the point where the properties of a material change compared to its bulk form.

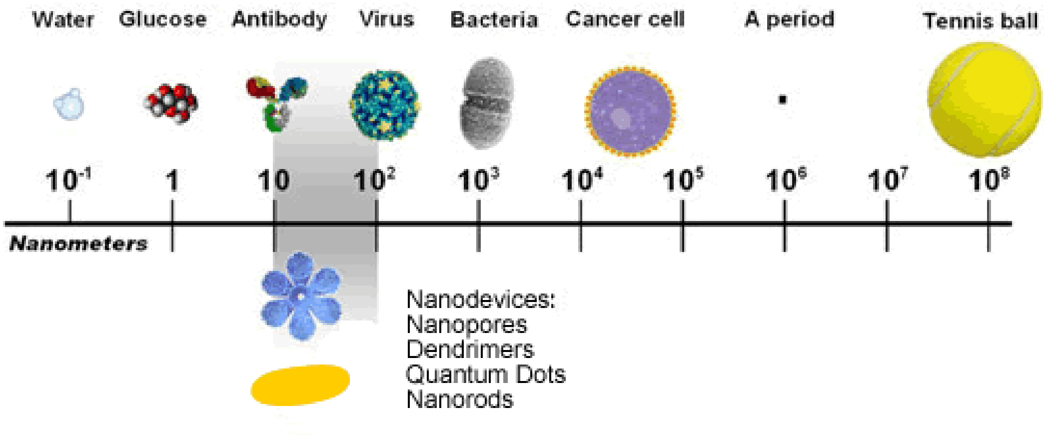

Figure 1.1: Example of materials within range of nanoscale [9].

A well-known material, zinc antimonides (Zn_4Sb_3) has been investigated widely, especially on obtaining figure of merit, thermal conductivity, Seebeck coefficient and electrical conductivity as those variables reflect to thermoelectric properties. Moreover, Zn_4Sb_3 is one of the cheapest materials known [10]. In recent paper, Zn_4Sb_3 would be a potential replacement for lead telluride as lead telluride is not environmental friendly [11]. However, there is still insufficient effort attempted by current researchers to do more investigation or improvement on Zn_4Sb_3 thin film properties, compared to bismuth telluride (Bi_2Te_3) thin film. Generally, the investigations are more on bulk form. To the best of our knowledge, scope of research such as determination of Zn_4Sb_3 thin film on surface morphology properties is still lack of exploration indeed.

In a basic Radio Frequency (RF) magnetron sputtering thin film deposition method, normally there are four main parameters that considered to be optimized, which are substrate temperature, deposition time, argon flow rate and RF power. These generated parameters would effect on chamber pressure, plasma induction and formation of the nanostructured thin film during deposition. Compared to other method, RF magnetron sputtering method is relatively easy-operated and produced narrow similar nanostructure size distribution. The magnetron is referred to magnet effect, which is been applied in order to minimize electron loss during sputtering.

In this research, a single sputtering target with 99.99% of Zn_4Sb_3 stoichiometric is utilized during deposition. Zn_4Sb_3 thin films are deposited with variation of growth parameters in order to obtain expected results. Once deposited, the Zn_4Sb_3 thin films are characterized by X-Ray Diffraction (XRD), Energy Dispersive X-ray (EDX), Field Emission Scanning Electron Microscopy (FESEM) and Atomic Force Microscopy (AFM) in order to gather information on surface morphology properties. The characterization would reveal information regarding the formation of nanostructure, uniformity and density of the deposited thin films, grain size observed and elementary composition.

1.2 Problem Statements

There are several attempts on Zn_4Sb_3 thin film deposition prepared by magnetron sputtering techniques. Zhang *et al* [12] had reported that applying co-deposited or co-sputtering technique during deposition would ended up deposited thin film with Zn-rich. Based on Ping Fan *et al* [13], there is high exposure of impurity issue during co-sputtering and it is difficult to control it. In this research, the single sputtering target Zn_4Sb_3 with 99.99% of purity is utilized during single sputtering to avoid impurity issue and to obtain the right stoichiometric of deposited thin film.

Growth parameters such as substrate temperature, deposition time, argon flow rate and RF power are manipulated accordingly in order to obtain wide range trend of results. Surface morphology properties of Zn_4Sb_3 thin films are characterized as it would reveal the relation between the growth parameters and variance of morphology formation, besides indicating the integrity of the sputtering mechanism itself. Based on previous research, there a lot of attempts to obtain thermoelectric properties of Zn_4Sb_3 thin film, as it one of the prominent thermoelectric material [12, 14, 15]. According to Locklin *et al* [16], the charge transport could be improved by tuning the density of deposited material, shape of nucleation and growth parameters of the thin film. In this research, the effect of growth parameters on surface morphology of Zn_4Sb_3 thin film is investigated.

Even though the effect of growth parameters on deposited thin film has been conducted deeply, there is still vast of information that can be revealed as it influences a wide range of effect. To the best of our knowledge, there is still lack of effort reported to obtain nanostructured Zn_4Sb_3 thin films through RF magnetron sputtering with optimized growth parameters. Zhang *et al* [12] reported that smaller dimension of structure would effect on boundary scattering, as a result it would increase figure of merit. In this research, the nanostructured of Zn_4Sb_3 thin films are deposited with optimized growth parameters.

1.3 Objectives of Research

This research aims to:-

1. deposit Zn_4Sb_3 thin films by RF magnetron sputtering for various growth parameters (substrate temperature, time deposition, argon flow rate and RF power)
2. determine the effect of growth parameters on surface morphology of Zn_4Sb_3 thin films

1.4 Scope of Research

In order to meet above objectives, this research is carried out as follow:-

1. Preparation of RF magnetron sputtering experimental setup.
2. Deposition of Zn_4Sb_3 thin films prepared by RF magnetron sputtering using a single sputtering target.
3. Characterization on surface morphology properties of nanostructured Zn_4Sb_3 thin films using XRD, EDX, FESEM and AFM.

1.5 Outline of Thesis

This thesis is consisting of five chapters. The first chapter describes an overview of research background and the significance of this research. The second chapter explains the previous research that had been conducted, besides contains the basic theory and summary of literatures related to this research. The third chapter presents the experimental setup, substrate preparation and samples characterization. The fourth chapter covers the experimental results obtained with detail analysis. The fifth chapter summarizes the conclusion of the research and also includes recommendation for future work.

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